इंटरनेट मानक

Disclosure to Promote the Right To Information

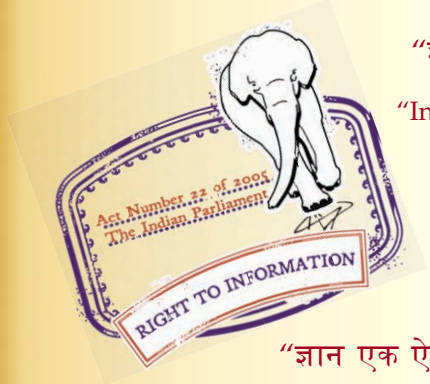
Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

"जानने का अधिकार, जीने का अधिकार"
Mazdoor Kisan Shakti Sangathan
"The Right to Information, The Right to Live"

"पुराने को छोड नये के तरफ"
Jawaharlal Nehru
"Step Out From the Old to the New"

IS 11774 (1986): Method for determination of dichlorvos residues in food commodities [FAD 1: Pesticides and Pesticides Residue Analysis]

"ज्ञान से एक नये भारत का निर्माण"
Satyanarayan Gangaram Pitroda
"Invent a New India Using Knowledge"



"ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है"
Bhartṛhari—Nītiśatakam
"Knowledge is such a treasure which cannot be stolen"

BLANK PAGE

IS : 11774 - 1986
(Reaffirmed 2003)

Indian Standard

METHOD FOR DETERMINATION OF DICHLORVOS RESIDUES IN FOOD COMMODITIES

UDC 664 : 543 : 632·951·028 DIC

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 2

February 1987

Indian Standard

METHOD FOR DETERMINATION OF DICHLORVOS RESIDUES IN FOOD COMMODITIES

Pesticides Residue Analysis Sectional Committee, AFDC 56

Chairman

DR H. L. BAMI
Bangalow No. A, Malkaganj, Delhi

Members	*Representing*
SHRI T. K. ADHYA	Central Rice Research Institute, Cuttack
SHRI E. A. ALMEIDA SHRI F. QUADROS (*Alternate*)	Hindustan Ciba-Geigy Ltd, Bombay
SHRI K. D. AMRE Da J. C. VERMA (*Alternate*)	National Organic Chemical Industries Ltd, Bombay
Da M. D. AWASTHI	Indian Institute of Horticultural Research (ICAR), Bangntore
DEAN DR R. P. SRIVASTAVA (*Alternate*)	Mohantal Sukhadia University, Udaipur
DR K. C. GUHA SHRI P. K. DHINGRA (*Alternate*)	Central Food Laboratory, Calcutta; *and* Central Committee for Food Standards, New Delhi
DR R. C. GVPTA DR (SHRIMATI) G. MUKHERJEE (*Alternate*)	Directorate of Plant Protection, Quarantine and Storage (Ministry of Agriculture and Rural Development), Faridabad
DR S. S. GUPTE DR P. V. RAIKAR (*Alternate*)	Bayer India Ltd, Thane
Da S. K. HANDA	Indian Agricultural Research Institute (ICAR), New Delhi
DR V. N. IYER DR R. SUNDARARAJAN (*Alternate*)	Coromandel Indag (P) Ltd, Madras
DR R. L. KALRA DR R. P. CHAWLA (*Alternate*)	Department of Entomology, Punjab Agricultural Univeroity, Ludhiana
DR KALYAN SINGH	C. S. A. University of Agriculture and Technology, Kanpur
DR J. C. MAJUMDAR DR V. SRINIVASAN (*Alternate*)	Pesticides Association of India, New Delhi
DR M. S. MITHYANTHA Da K. SIVASANKARAN (*Alternate*)	Rallis Agrochemical Research Station, Bangalore

(*Continued on page* 2)

(*Continued from page* 1)

Members	*Representing*
DR A. L. MOOKERJEE	Cyanamid India Ltd, Bombav
DR S. Y. PANDEY	Union Carbide India Ltd, New Delhi
SHRI R. RAJGOPAL	National Institute of Communicable Diseases, Delhi
SHRI G. C. JOSHI (*Alternate*)	
Da M. S. SASTRY	Indian Veterinary Research Institute (ICAR), Izatnagar
DR T. K. VERMA (*Alternate*)	
DR T. D. SETH	Industrial Toxicology Research Centre (CSIR), Lucknnw
DR R. K. SETHI	Indofil Chemicals Ltd, Thane
DR R. SUNDARARAJAN	Fredrick Institute of Plant Protection and Toxicology, Padappai
DR K. VISHESWARIAH	Central Food 'Technological Research Institute (CSIR), Mysore
DR J. R. RANGASWAMY (*Alternate*)	
SHRI T. PURNANANDAM, Director (Agri & Food)	Director General, ISI (*Ex-officio Member*)

Secretary

SHRI LAJINDER SINGH
Joint Director (Agri & Food), ISI

Indian Standard

METHOD FOR DETERMINATION OF DICHLORVOS RESIDUES IN FOOD COMMODITIES

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 18 September 1986, after the draft finalized by the Pesticides Residue Analysis Sectional Committee had been approved by the Agricultural and Food Products Division Council.

0.2 Dichlorvos is used in agriculture for the control of insect pests. Assessment of residues is, therefore, an important step in safeguarding human health.

0.3 In the preparation of this standard, due consideration has been given to the limits of dichlorvos laid under the provisions of *Prevention of* ***Food Adulteration Rules,*** 1965 and the test methods are sensitive to the prescribed levels of residues.

0.4 This standard will enable the health authorities and others engaged in the field to follow a uniform test procedure for the estimation of dichlorvos residues in various food commodities.

0.5 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes the gas chromatographic method (GLC) for the determination of dichlorvos residues in food commodities.

2. QUALITY OF REAGENTS

'2.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1977†) shall be employed in tests.

NOTE — 'Pure **chemicals** shall mean chemicals that do not contain impurities which affect the results of analysis.

*Rules for rounding off numerical values (*revised*).

†Specification for water for general laboratory use (*second revision*).

3. SAMPLING

3.1 The representative samples for the purposes of estimating dichlorvos residues in foods shall be drawn in accordance with IS : 11380 (Part 1)-1985*.

4. PREPARATION OF THE LABORATORY SAMPLE, CLEAN UP AND EXTRACTION

4.1 Apparatus

4.1.1 ***Waring Blender or Equivalent***

4.1.2 Kuderna Danish Evaporative Concentrator

4.1.3 Water-Bath

4.1.4 Chromatographic Column — ***25*** cm long and 2 cm i.d.

4.2 Reagents

4.2.1 Methylene Chloride — AR grade, glass redistilled.

4.2.2 n-Hexane — AR grade, glass redistilled.

4.2.3 Ethyl Acetate — adsorption grade, glass redistilled.

4.2.4 Sodium Sulphate — anhydrous.

4.3 Extraction

4.3.1 ***Fruits and Vegetables*** — Shred the entire laboratory sample. Macerate a subsample of 25 g with 100 ml of methylene chloride using the waring blender. Filter the suspension under slight vacuum through a 6-cm buchner funnel layered with suitable filter paper. Rinse, jar and filter cake twice with 50 ml portions of methylene chloride. Transfer the methylene chloride extract to a separatory funnel, and drain off the organic layer into 500-ml erlenmeyer flask. Dry with 4-5 g of anhydrous sodium sulphate, and filter through a folded paper into a 500-ml round-bottom flask. Concentrate the methylene chloride solution to about 20 ml and proceed as described under 4.4.

4.3.2 ***Extraction of Oily Crops*** — Place 25 g of milled grain into a soxhelt thimble and extract with 300 ml of methylene-chloride in a soxhlet apparatus for 4 hours, using a 500-ml round bottom flask, Concentrate the extract to approximately 20 ml by using the rotating evaporator (35-40°C) and add 50 ml of hexane. Concentrate again, and add a further portion of 50 ml hexane. Concentrate second time to a volume of 10-15 ml and transfer the hexane extract to a 50-ml volumetric flask. Wash the evaporation flask twice with small portions of hexane, add the washings to the extract and make up to volume with hexane.

*Methods of sampling for determination of pesticides residues : Part 1 Agricultural and food commodities.

4.4 **Clean-Up** — Extract the hexane solution (50 ml) four times with 100 ml of distilled water in a 250-ml separatory funnel. Combine the **aqueous extracts** and re-extract dichlorvos from the water with two 100-ml portions of ethyl acetate using 1-litre separatory funnel. Collect the **organic** solutions in an erlenmeyer flask, dry with 4-5 g anhydrous sodium sulphate, and filter through a folded paper into a round-bottom flask. Wash the erlenmeyer flask and the sodium sulphate with 50 ml of hexane, which portion is added to the ethyl acetate extract. Concentrate on the rotating evaporator (35-40°C) to a volume of about 5 ml, and transfer the concentrate to a 10-ml glass-stoppered graduated test tube. Rinse evaporation flask with 3-4 ml of hoxane, add the washing to the concentrate, and make up to 10 ml.

5. GAS **CHROMATOGRAPHIC METHOD**

5.1 The extracted dichlorvos is measured gas chromotographically using a NPD detector or a flame photometric detector (phosphorus specific). The content of dichlorvos **is** determined by comparing the response with reference standard of dichlorvos.

5.1.1 Gas *Chromatograph* — A gas chromatograph equipped with a NPD detector or a flame photometric detector operated under the following suggested parameters. These parameters may be varied according to the available facilities provided standardization is done:

Column	Glass, 2 ml long and 2 mm i.d. packed with 10 percent 0 *V* 2 10 on Gas Chrom Q (80-100 mesh)	
Column oven temperatute	130°C	
Injection port temperature	200°C	
Detector temperature	200°C	
Carrier gas and flow rate	Nitrogen Hydrogen Air	40 7 15-20 } ml per minute 150 J
Retention time	5 mts	
Chart speed	5 mm/mt	
Attenuation	100 x 1	

5.2 **Procedure** — Inject suitable aliquot (1 μl) into the column using a microlitre syringe. Identify the peak by its retention time and measure the peak area. For reference standard prepare hexane solutions containing one to ten ppm of dichlorvos.

5.3 Calculation

$$\text{Dichlorvos residue, } \mu g/g \text{ (ppm)} = \frac{A_1 \times V_2 \times V_3 \times C \times f}{A_2 \times V_1 \times M}$$

where

A_1 = peak height/area of the sample;

V_2 = volume, in μl, of standard dichlorvos injected;

V_3 = total volume, in ml, of sample solution;

C = concentration, in $\mu g/g$, of standard dichlorvos solution;

f = recovery factor = $\frac{100}{\text{percent mean recovery}}$,

A_2 = peak height/area of the dichlorvos standard;

V_1 = volume of sample in μl injected; and

M = mass, in g, of sample taken for analysis.